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NIKEL(II) NING AMMIN-[N-[6,6,7,7,8,8,8-GEPTAFTOR-5-GIDROKSI-2,2-DIMETILOKT-4-EN-3-ILIDEN]BENZENKARBOGIDRAZONAT] KOMPLEKSINING SINTEZI VA TUZILISHI

Annotatsiya

Mazkur ishda ftorlangan gidrazon ligand asosida nikel(II) ning yangi amminli kompleks birikmasi sintez qilindi. Boshlang‘ich ligandning tuzilishi IQ spektroskopiya usuli yordamida tasdiqlandi. Olingan nikel(II) kompleksining molekulyar va kristall tuzilishi esa monokristall rentgen difraksion tahlil yordamida aniqlandi. Tadqiqot natijalariga ko‘ra, ligand nikel(II) ioniga donor atomlari orqali koordinatsiyalanib, barqaror kompleks hosil qilishi ko‘rsatildi.

Kalit so‘zlar: nikel(II) kompleksi, gidrazon, ftorlangan ligand, ammin kompleksi, rentgenstruktur tahlil, koordinatsion birikma, yutulish chiziqlari, benzogidrazid, valent tebranishlar.

SYNTHESIS AND STRUCTURE OF AMMINE-[N-[6,6,7,7,8,8,8-HEPTAFLUORO-5-HYDROXY-2,2-DIMETHYLOCT-4-EN-3 YLIDENE]BENZENECARBOHYDRAZONAT]NICKEL(II)

Annotation

In this work, a new ammine nickel(II) complex compound based on a fluorinated hydrazone ligand was synthesized. The structure of the initial ligand was confirmed by IR spectroscopy. The molecular and crystal structure of the obtained nickel(II) complex was determined by single-crystal X-ray diffraction analysis. According to the research results, it was shown that the ligand coordinates to the nickel(II) ion through donor atoms and forms a stable complex.

Keywords: nickel(II) complex, hydrazone, fluorinated ligand, amine complex, X-ray structural analysis, coordination compound, absorption lines, benzohydrazide, valence vibrations.

СИНТЕЗ И СТРУКТУРА АММИН-[N-[6,6,7,7,8,8,8-ГЕПТАФТОР-5-ГИДРОКСИ-2,2-ДИМЕТИЛОКТ-4-ЕН-3-ИЛИДЕН]БЕНЗОЛКАРБОГИДРАЗОНАТ] НИКЕЛЯ(II)

Аннотация

В данной работе был синтезирован новый амминный комплекс никеля(II) на основе фторированного гидразонного лиганда. Структура исходного лиганда была подтверждена методом ИК-спектроскопии. Молекулярное и кристаллическое строение полученного комплекса никеля(II) было установлено методом монокристального рентгеноструктурного анализа. По результатам исследования показано, что лиганд координируется к иону никеля(II) через донорные атомы с образованием устойчивого комплекса.

Ключевые слова: Комплекс никеля(II), гидразон, фторированный лиганд, аминный комплекс, рентгеноструктурный анализ, координационное соединение, линии поглощения, бензогидразид, валентные колебания.

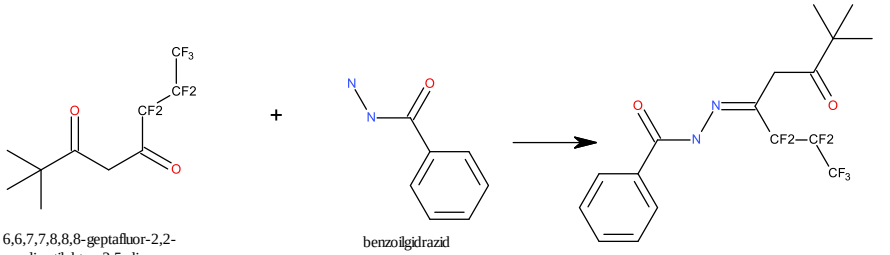
Kirish. Koordinatsion kimyo sohasida o‘tish metallari asosidagi kompleks birikmalarni sintez qilish va ularning tuzilishini o‘rganish muhim ilmiy yo‘nalishlardan biri hisoblanadi. Bunday birikmalar nafaqat nazariy jihatdan, balki amaliy jihatdan ham qiziqish uyg‘otadi. Metall komplekslar kataliz, analitik kimyo, materialshunoslik, biologik faol moddalarning modellashtirilishi kabi ko‘plab sohalarda muhim ahamiyatga ega [1].

Adabiyotlar tahlili. Azot va kislorod donor atomlariga ega bo‘lgan organik ligandlar o‘tish metallari bilan mustahkam koordinatsion birikmalar hosil qilishi bilan ajralib turadi. Xususan, gidrazon va karbogidrazid hosilalari koordinatsion kimyoda keng qo‘llaniladigan ligandlar qatoriga kiradi. Ularning tarkibida azometin guruhi (>C=N–), karbonil yoki enol fragmentlari, shuningdek proton donor funksional guruhlarning mavjudligi metall ionlari bilan turli tipdagi koordinatsion bog‘lar hosil bo‘lishiga imkon yaratadi[2].

So‘nggi yillarda ftorlangan ligandlar asosidagi kompleks birikmalarga bo‘lgan qiziqish yanada ortib bormoqda. Bunga sabab ftor atomlarining kuchli elektronakseptor xususiyatga egaligi bo‘lib, ular ligandning elektron tuzilishi, kislotali-asosli xossalari, kompleks hosil qilish qobiliyati hamda umumiy barqarorligiga sezilarli ta’sir ko‘rsatadi. Ftorlangan zanjir yoki trifluormetil fragment tutgan ligandlar asosida olingan komplekslar odatda yuqori termik va kimyoviy barqarorlikka ega bo‘ladi [2-4].

Mazkur ishning maqsadi Nikel(II) ning ammin-[N-[6,6,7,7,8,8,8-geptaftor-5-gidroksi-2,2-dimetilokt-4-en-3-iliden] benzenkarbogidrazonat] kompleksining sintezi va tuzilishi kompleksini sintez qilish, boshlang‘ich ligandning spektral tavsifini berish hamda olingan kompleksning molekulyar va kristall tuzilishini monokristall rentgen difraksion tahlil asosida aniqlashdan iborat.

Tajriba qismi. Gidrazon ligandining (H_2L) sintezi. 1.7556g (12.9 mmol) benzoilgidrazid 50 ml etanolda to‘liq eritildi. Hosil bo‘lgan eritmaga 3 ml 2,2-dimetil-6,6,7,7,8,8,8-geptafluoro-3,5-oktandion tomchilab qo‘shildi. Reaksion aralashma teskari sovutgich ostida 2 soat davomida qaynatildi.



6,6,7,7,8,8,8-geptafluor-2,2-dimetiloktan-3,5-dion

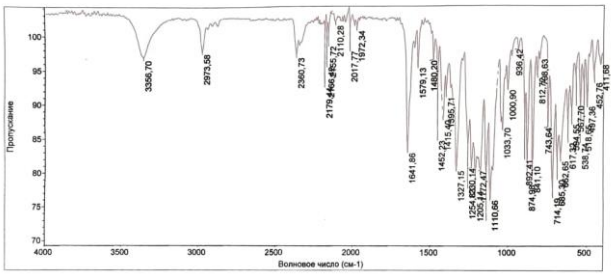
benzoilgidrazid

(Z)-N'-(1,1,1,2,2,3,3-geptafluoro-7,7-dimetil-6-okso butan-4-iliden)benzogidrazid

1 - rasm. (Z)-N'-(1,1,1,2,2,3,3-geptafluoro-7,7-dimetil-6-okso butan-4-iliden) benzogidrazidning hosil bo‘lish reaksiyasi

Reaksiya yakunlangach, eritma xona haroratigacha sovutildi va sekinlik bilan bug‘latildi. Natijada oq rangli, yarim shaffof kristall modda ajralib chiqdi. Olingan ligand etanolda qayta kristallab tozalandi va eksikatorda quritildi.

Gidrazon ligandining (H_2L) IQ spektri tahlili



2-rasm. (Z)-N’-(1,1,1,2,2,3,3-geptafluoro-7,7-dimetil-6-oksooktan-4 iliden) benzogidrazid molekulasining IQ spektri

Ushbu tahlilda sintez qilingan yangi organik birikma - (Z)-N’-(1,1,1,2,2,3,3-geptafluoro-7,7-dimetil-6-oksooktan-4-iliden) benzogidrazid molekulasining tuzilishi infraqizil (IQ) spektroskopiya usuli yordamida o‘rganildi. Spektrdagi yutilish chiziqlarining chastotalari molekuladagi xarakterli funksional guruhlar va bog‘larning tebranishlariga to‘liq mos keladi.

Molekuladagi yuqori chastotali yutilish chiziqlari vodorod atomi ishtirokidagi bog‘larning valent tebranishlarini ifodalaydi. $3356.70\,\text{cm}^{-1}$ sohada kuzatilgan keng va intensiv yutilish chiziqlari gidrazid guruhidagi N-H bog‘i valent tebranishiga (υ_{N-H}) tegishli. Bu molekulada ikkilamchi amin guruhining saqlanib qolganligini va molekulalararo vodorod bog‘lanishlar mavjudligini ko’rsatadi. $2973.58\,\text{cm}^{-1}$: Bu cho‘qqi alifatik C-H bog‘larining (υ_{C-H}) valent tebranishlariga mos keladi. U molekula tarkibidagi tert-butil guruhining metil ($-CH_3$) va zanjirdagi metilen ($-CH_2-$) guruhlari mavjudligini tasdiqlaydi[5-6].

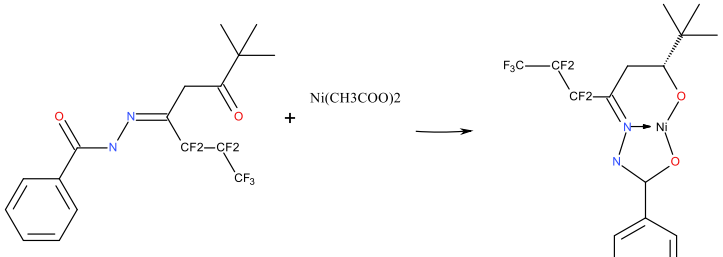
$1641.86\,\text{cm}^{-1}$ soha spektrdagi eng xarakterli cho‘qqilardan biri bo‘lib, u karbonil guruhi ($\upsilon_{C=O}$) valent tebranishiga tegishli.

$1327.15 - 1110.66\,\text{cm}^{-1}$ intensiv yutulish chiziqlar seriyasi geptaforpropil **($-C_3F_7$)** guruhidagi $C-F$ bog‘larining valent tebranishlariga (υ_{C-F}) mos keladi.

$743.64 - 714.19\,\text{cm}^{-1}$ cho‘qqilar monosubstitutsiyalangan benzol halqasidagi $C-H$ bog‘larining tekislikdan tashqari deformatsion tebranishlarini (γ_{C-H}) ko‘rsatadi. Bu benzogidrazid qoldig‘idagi aromatik halqaning bir o‘rinbosarli ekanligini tasdiqlovchi asosiy dalildir.[7-8]

IQ-spektroskopik tahlil natijalari shuni ko‘rsatadiki, barcha asosiy yutilish polosalari ($3356\,\text{cm}^{-1}$ da $N-H$, $1641\,\text{cm}^{-1}$ da $C=O/C=N$, $1100-1300\,\text{cm}^{-1}$ sohasidagi intensiv $C-F$ va aromatik halqa signallari) o‘rganilayotgan (Z)-N’-(1,1,1,2,2,3,3-geptafluoro-7,7-dimetil-6-oksooktan-4 iliden) benzogidrazid molekulasining kimyoviy tuzilishini to‘liq tasdiqlaydi. Spektral ma’lumotlar molekulada gidrazon bog‘lanishi va kuchli elektron akseptor xususiyatga ega bo‘lgan perftoralifatik zanjir mavjudligidan dalolat beradi [9].

Nikel(II) ammin kompleksining sintezi. Nikel(II) ammin kompleksini sintez qilish uchun ligand va metall tuzi 1:1 molyar nisbatda olindi. Dastlab, nikel(II) atsetat tetragidrati konsentrlangan ammiak eritmasida (25%) to‘liq eritildi (to‘q ko‘k rangli eritma hosil bo‘lguncha). Tayyorlangan nikel ammiakat eritmasi ligandning etanoldagi eritmasiga doimiy aralashtirish ostida qo‘shildi. Aralashma xona haroratida 1 soat davomida aralashtirildi. Hosil bo‘lgan kompleks birikma cho‘kma holida ajratildi, distillangan suv va sovuq etanol bilan yuvildi hamda vakuum ostida quritildi.

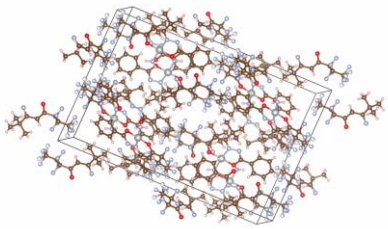


(*Z*)- *N*'-(1,1,1,2,2,3,3-heptafluoro-7,7-dimethyl-6-oxooctan-4-ylidene)benzohydrazide

ammin-{N-[6,6,7,7,8,8,8-geptaftor-5-(gidroksi)-2,2-dimetilokt-4-en-3-iliden]benzenkarbogidrazonato}-nikel

3-rasm. Nikel(II) ning ammin-[N-[6,6,7,7,8,8,8-geptaftor-5-gidroksi-2,2-dimetilokt-4-en-3-iliden]benzenkarbogidrazonat] kompleks birikmasining hosil bo‘lish reaksiyasi

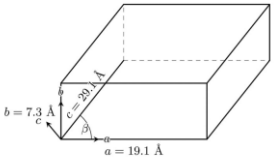
Nikel(II) ning ammin-[N-[6,6,7,7,8,8,8-geptaftor-5-gidroksi-2,2-dimetilokt-4-en-3-iliden]benzenkarbogidrazonat] kompleksining kristall strukturasi rentgen-strukturaviy tahlil (RST) usuli yordamida o‘rganildi.



4-rasm. Nikel(II) ning ammin-[N-[6,6,7,7,8,8,8-geptaftor-5-gidroksi-2,2-dimetilokt-4-en-3 iliden] benzenkarbogidrazonat] kompleks birikmasining 3D fazoviy tuzilishi.

Kristall panjara parametrlari I 2/a fazoviy guruhiga muvofiq aniqlangan. Monoklin panjaraning β burchagi 94.050(4)∘ ga teng bo’lib, bu standart monoklinik og‘ishni ko’rsatadi. Z=8 qiymati elementar yacheyka ichida sakkizta mustaqil kompleks birligi mavjudligini anglatadi[10-11]

Strukturaning aniqlik darajasini belgilovchi R-faktor 5.4% ni tashkil etadi.



5-rasm. Monoklin elementar yacheyka parametrlari sxemasi. B=94.05°, α=γ=90°

Ushbu ko‘rsatkich olingan strukturaviy modelning eksperimental rentgen difraksiyasi ma’lumotlariga yuqori darajada mos kelishini va atomlar koordinatalarining ishonchliligini tasdiqlaydi.

Xulosa. Kompleks birikmaning molekulyar va kristall tuzilishi monokristall rentgen difraksiyasi usuli yordamida aniqlanib, taklif etilgan koordinatsion tuzilma tasdiqlandi. Rentgen-strukturaviy tahlil natijalariga ko‘ra, birikma ma’lum kristall tizimida kristallanadi va tegishli fazoviy guruhga mansubdir.

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